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(21) International Application Number: PCT/EP99/00325 (22) International Filing Date: 19 January 1999 (19.01.99) (30) Priority Data: 98300792.3 4 February 1998 (04.02.98) EP (71) Applicant (for AU BB CA CY GB GD GH GM IE IL KE LC LK LS MN MW NZ SD SG SL SZ TT UG ZW only): UNILEVER PLC (GB/GB); Unilever House, Blackfriars, London EC4P 4BQ (GB). (71) Applicant (for all designated States except AU BB CA CY GB GD GH GM IE IL IN KE LC LK LS MN MW NZ SD SG SL SZ TT UG US ZW): UNILEVER N.V. (NL/NL); Weena 455, NL-3013 AL Rotterdam (NL). (71) Applicant (for IN only): HINDUSTAN LEVER LIMITED (IN/IN); Hindustan Lever House, 165/166 Backbay Reclamation, Mumbai 400 20, Maharashtra (IN). (72) Inventors; and (75) Inventors/Applicants (for US only): BOLZONI, Giuseppe, Vincenzo (IT/IT); Lever Development Centre, Via Lever Gibbs, 3/a, I-26841 Casalpusterlengo (IT). MORRIS, Ronald, Meredith (GB/HU); Unilever Magyarorszag KFT,		Devai u. 26-28, H-1134 Budapest XIII (HU). PUERARI, Francesco, Maria (IT/IT); Lever Development Centre, Via Lever Gibbs, 3/a, I-26841 Casalpusterlengo (IT). TUMMILOLO, Roberto (IT/IT); Lever Development Centre, Via Lever Gibbs, 3/a, I-26841 Casalpusterlengo (IT). (74) Agent: TANSLEY, Sally, Elizabeth; Unilever PLC, Patent Dept., Colworth House, Sharnbrook, Bedford MK44 1LQ (GB). (81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published With international search report.	
(54) Title: LAVATORY CLEANSING COMPOSITIONS			
(57) Abstract			
A lavatory cleansing block comprising: a) a particulate bleaching agent or precursor therefore, and b) a dyestuff on a particulate, non-bleaching carrier.			

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LAVATORY CLEANSING COMPOSITIONS

Technical Field

5 This invention is concerned with improvements in and relating to lavatory cleansing compositions. More particularly, the invention is concerned with solid lavatory cleansing compositions which may be used to impart cleansing and/or other components to the flush water of a lavatory or
10 urinal by placement of the composition in the cistern of the lavatory or urinal.

Background to the Invention

15 The use of solid slow-release compositions to impart cleansing and/or other components to the flush water of a lavatory or urinal is well known. Such compositions may, for example, be immersed in the water of a lavatory cistern either free-standing form or in containerised form (i.e., in
20 a dispensing container which allows for release of components of the solid composition in solution in water in a more or less metered fashion, on flushing of the cistern).

Alternatively, the solid composition may be held under the
25 rim of a toiler, in a suitable holder, for intermittent contact of the solid material with flush water.

For convenience these two approaches will be referred to as 'cistern-blocks' and 'rim-blocks' herein and known
30 collectively as 'toilet-blocks'. Such blocks produce foam, often produce a pleasing odour and can have germicidal properties.

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Solid lavatory cleansing compositions typically comprise a surfactant component, generally together with one or more fillers or builders (e.g. inorganic salts such as sodium sulphate, sodium chloride etc). Commonly, such compositions also contain a perfume, a dyestuff and, frequently, dissolution retarding material.

In recent years it has become commonplace for toilet blocks to contain a halogen release agent or other bleaching agent. From EP 0206725 it is clear that halogen release agents are, by their nature, powerful chemically reactive species, serving as halogenating or oxidising agents and it is consequently difficult to incorporate bleach-sensitive components such as perfumes and dyestuffs into blocks which contain bleaching agents.

It is, however, desirable to be able to determine rapidly when bleach is present in a block and thereby distinguish it from blocks which do not contain bleach. Several possible ways of providing a visual indication that a block contains bleach can be envisaged: one might for example make the block a particular shape or mark the surface of the block with embossed pattern. While it would be desirable to incorporate a colouring agent into a bleach-containing block this is not generally possible due to the interaction of the colour and the bleaching agent.

In the related art of liquid toilet bleaches, it has been suggested to colour formulations by means of suspended pigment particles. These retain their colour in the presence of the bleaching agent and provide a visual means of identifying where a product has been used as well as

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rough indication of dilution. Unfortunately, a quality sanitary porcelain is prone to form on its surface into which the pigment particles providing a clear visible indication that surface are present. As a result, the use of pigments has not met with commercial success.

tion of the Invention

mined that improved toilet blocks can be which comprise: a particulate bleaching agent (or) and a dyestuff on a particulate carrier other ing agent. This invention is of particular ed in in-cistern blocks.

the present invention provides a slow-release nsing block comprising:

ulate bleaching agent or precursor therefore,

ff on a particulate, non-bleaching carrier.

g the bleaching agent and the dyestuff in this possible to produce a block which provides a ation that a bleaching agent is present in the tinct speckled pattern thus the present ther provides the use of coloured speckles tory cleansing block to indicate the presence g agent.

Detailed Description of the Invention

- 5 In order that the invention may be further understood the following description details preferred and optional features of the invention.

Dyestuff and Carrier

10

Typically, blocks according to the present invention comprise 1-15% wt, more preferably 2-10% of a dyestuff on a carrier.

- 15 Preferably the dye stuff is water dispersible, more preferably the dye is water soluble. Dyes that are blue or green are particularly suitable for indicating that bleach is present. We have determined that 'Sudangelb 150' (TM) ex. Sandoz is an acceptable colouring agent as are Colanyl
- 20 Green (TM) ex Hoechst P. Green (CI 74260) and Dispers Blue (TM) ex BASF. Particularly preferred colouring agent include pigments such as pigment blue 15 having a colour index of 74160.

- 25 Typically the dye stuff is present from 0.05 to 0.5 wt % of the total weight of the dyestuff/carrier granule.

- It is preferred if the particulate non-bleaching carrier is water soluble. Water soluble in the context of the
- 30 invention is a solubility of at least 1g per 100 cc of water at 20°C.

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It is preferred if the particulate non bleaching carrier is selected from sodium carbonate, sodium sulphate, magnesium sulphate, or sodium chloride, particularly preferred is sodium carbonate.

5

The particulate non bleaching carrier is preferably present from 93% to 99% wt% of the dyestuff/carrier granule.

10

It is advantageous if the dyestuff/carrier granule further comprises a binder such as a polycarylate a preferred binder is an acrylic maleic copolymer. The binder is preferably present from 0.1 to 1% wt of the dyestuff/carrier granule.

15

The dyestuff/carrier granules preferably have a particle size distribution between 100 and 1000 microns and a bulk density of 900 and 1150 g/l.

Bleaching Agents

20

Typically the blocks comprise up to 50% by weight of an at least sparingly water soluble bleaching agent. Typically levels of bleaching agents are 2-30% wt on product. For the purposes of the present specification the term bleaching agent is used to mean both a bleaching agent and a precursor which produces a bleaching agent unless the context demands otherwise.

25

30

Suitable bleaching agents active-halide and active-oxygen bleaching agents, particularly the so-called 'halogen release agents'.

Chlorine bleaching agents are preferred. Suitable water-soluble, active chlorine, bleaching agents used in

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accordance with the invention include chlorinated cyanurates, phthalimides, p-toluence sulphonamides, azodicarbonamides, hydantoins, glycoluracils, amines and melamines. The alkali metal salts of cyanurates are preferred.

A particularly preferred bleaching agent is sodium dichlorocyanurate (NaDCCA). The bleaching agent is typically present in an amount of 10-30% and most preferably at around 25%. Oxidan DCN/WSG (TM) ex Sigma has been found to be a suitable bleaching agent.

Surfactants

Preferably, the composition will also contain a surfactant component which may be anionic or nonionic in nature. The surfactant serves to provide a cleansing and foaming effect.

Preferably, the surfactant component comprises one or more anionic surface active agents, optionally in combination with one or more nonionic surface active agents. Suitable anionic surface active agents include alkali metal or ammonium alkylaryl sulphonates (especially alkyl benzene sulphonates), alkane sulphonates, alkyl sulphates and sarcosinates.

We have determined that improved foaming properties are obtained by the use of a surfactant system which comprises primary alkyl sulphate (PAS) together with other anionic surfactants. Preferably the present invention provides a lavatory block comprising 1-15% wt (more preferably 1-5% wt) of a primary alkyl sulphate and 15-50%wt (more preferably 30-50%wt) of other anionic surfactants. We have determined

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that the use of this mixed surfactant system is advantageous in that it reduces the stickiness of the block during manufacture. The blocks with 1-5% PAS show improved wear characteristics.

5

Preferably the other anionic surfactants are sulphonates. Suitable sulphonates include alkyl benzene sulphonate (ABS). It is believed that the combination of relatively low levels of PAS together with higher levels of ABS promotes the foaming and the perfume delivery from the block. PAS is also believed to be environmentally more acceptable than alkyl benzene sulphonate.

10

Suitable nonionic surfactants include polyethoxylated fatty alcohols, polyethoxylated fatty acids, polyethoxylated alkyl phenols, amine oxides and ethylene oxide/propylene oxide block copolymers.

15

The total amount of surfactant when present, may lie within wide limits. In practice, the surfactant will generally be for 10 to 70% by weight of the composition, but more preferable that surfactant comprises from 20 to 50% by weight thereof.

20

25 Process Aids

As will be described in further detail below extrusion is the preferred method of manufacture. Optionally, the blocks according to the invention further comprise a processing aid to assist in extrusion. Suitable processing aids include oils (including both mineral and silicone oils), esters (other than those derived from ethylenically unsaturated carboxyl group containing monomers) and polybutene.

30

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One particularly suitable processing aid is an alkoxyated alcohol. It is preferred that the alkoxyated alcohol is an ethoxyated alcohol. The preferred level of alkoxyated alcohol is 0.75-2% wt. The preferred ethoxy chain length is 40-60 with an average ethoxy chain length of around 50 being preferred. Suitable materials include Empilan KM 50/KF (TM) ex. Albright & Wilson. Another suitable process aid is a PEG monostearate. PEG 4000 Monostearate (100%) ex DAC is a suitable raw material. It is believed that an effect of the process aid is to make the block harder and easier to cut from an extruded billet.

Fillers

Typically, blocks according to the present invention comprise 0 to 50% by weight of an inert non-polymeric and/or electrolyte filler. Preferably blocks contain 5-50%wt, more preferably 10-30% of filler.

Suitable fillers include one or more of urea, sodium, magnesium and calcium carbonates, sodium chloride, borax, talc and sodium, magnesium and calcium sulphates. Preferred ionic fillers include sodium sulphate. Preferred inert, non-polymeric fillers include calcium carbonate.

Typical levels of total filler range from 10-40%wt on product in total.

It has been found useful, for ease of formulation and to ensure complete solubility of in-cistern blocks, to employ 10-30%wt of an ionic filler as the sole filler present.

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For mixed filler systems, preferred levels of filler are 10-20%wt on product of ionic filler and 10-20% on product of inert filler. It is particularly preferred to use an approximately 50:50 mixture of sodium sulphate and calcium carbonate as the filler. For mixed filler systems typical levels in product are 15%wt of each of calcium carbonate and sodium sulphate.

Perfume

Preferably, the blocks comprise 2-15%wt of a hydrophobic oily liquid perfume. The blocks more preferably comprise, 2-10%, more preferably 3-6% wt of the perfume. Levels of around 4% wt perfume are particularly preferred. This oily perfume is typically of the kind described in the European patent application EP 167,210. It will be understood that the liquid oily perfume must be stable in the presence of the water-soluble, active chlorine, bleaching agent. Suitable oily perfumes can be easily selected by testing them in combination with the water-soluble, active chlorine, bleaching agent.

Examples of suitable bleach-stable perfumes are Verdeo 898, Bonanza 048 and Ponderosa 431 all ex IFF, and LB 132 ex Quest. Particularly preferred perfumes are Icebreaker Super Mod, Oxygen Supra Mod, Motebianco Supra and lemonfit Supra (all TM) ex Givaudan Roure. The most preferred perfume is Green Tank Harder (TM) ex. Givaudan Roure.

Minors

- 5 Minor components will generally be present but are optional. These include colouring agents, and/or whiteners. These materials should be chosen such that they are compatible with the bleaching agent and do not react therewith to a significant extent. Titanium dioxide is an acceptable
10 whitener. Levels of colouring agents and/or whiteners as typically below 5% wt.

Further enhancement of the product may be obtained by the additional use of chelating agent, sequestrant or water-
15 softening agent such as ethylene diamine tetra-acetic acid or a derivative thereof, nitrolotriacetic acid, phosphonates of polyphosphates, metasilicates, boroheptonates, s.s-ethylene-diamino disuccinate, dipicolinic acid, 2-phosphonobutane-1,2,4-tricarboxylic acid, or lower molecular
20 weight polymeric materials capable of inhibiting crystal growth. Further reducing agents, such as alkali metal metabisulphates may be present to assist in the reduction of staining due to metals such as iron.

- 25 An optional minor component is a foam-boosting surfactant. Suitable surfactants include amine oxides.

Polymers

- 30 A water-insoluble, gelling polymers may be used in compositions of the invention, these are polycarboxylic acids derived from one or more ethylenically unsaturated

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carboxyl group-containing monomers, especially ethylenically unsaturated carboxylic acids such as acrylic acid or maleic acid.

- 5 Suitable polymers include polyacrylic acid, polymaleic anhydride and copolymers of either of the aforementioned with ethylene, styrene and methyl vinyl ether.

- 10 Typical polymers are polyacrylic acid and acrylic acid/maleic acid copolymers. In practice the polymers are employed in the form of acids, but can also be employed as salts, e.g., alkali metal salts such as the sodium salt. The use of the polymer salts is advantageous if it is required to reduce dust production during formulation and
15 manufacture.

- If present it is preferred that the polymer is cross-linked. The cross-linked polyacrylate polymers are generally characterised as acrylic acid polymers which are cross-
20 linked with an additional monomer or monomers in order to exhibit an effective molecular weight of one to seven million g/mole. The average formula weight for a polymer sub-unit is preferably of the order of 60-120 g/mole.

- 25 The polymer is suitably present in an amount of from 0.5 to 20% by weight, more preferably from 1 to 5% by weight, most preferably around 2-3% by weight thereof. Polygel DB (TM) ex 3V Sigma, a cross-linked high molecular weight polyacrylate, has been found to be a suitable material at an
30 inclusion level of around 2-3%wt.

Process

5 Compositions in accordance with the invention may be produced by a variety of routes. For example, they may be prepared by a so-called "hot-melt" process comprising melting the fusible constituents of the block either alone and then adding other components in admixture with non-
10 fusible components, and subsequently casting the melt into moulds. More preferably, however, compositions in accordance with the invention are formed into the desired final shape by a compression technique, i.e., a technique involving the steps of forming a mixture of the ingredients
15 of the compositions and then compressing that mixture into the desired shape.

An especially preferred process is an extrusion process in which the mixture of the components is extruded into a solid
20 bar or rod which is subsequently cut into pieces of the desired size. In this connection, it may be noted that when the compositions of the invention are used as free-standing lavatory cleansing blocks, these suitably have a weight from 30 to 150 gms. When extruding a solid composition it is
25 generally advantageous, as noted above, that some lubricant component or process aid be present to facilitate extrusion.

Compositions in accordance with the invention may also be formed into the final desired shape by a tabletting
30 technique.

As used herein the term block is not intended to limit the shape of the eventual product. For cistern blocks the rod

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is cut into lengths which are short relative to their diameter.

5 **Preferred Compositions**

Preferred embodiments of the invention provide a slow release lavatory cleansing block comprising:

- 0 a) 2-30% wt of a halogen release agent,
b) 2-15% wt of an oily liquid perfume, and
c) 1-5% wt of a water dispersible dyestuff on a particulate, non bleaching carrier.

15 Particularly preferred compositions are:

- a) 1-5% wt primary alkyl sulphate
b) 30-50% wt alkyl benzene sulphonate
20 c) 10-30% wt NaDCCA
d) 10-30% wt sodium sulphate, calcium carbonate or a mixture thereof,
e) 2-10% wt oily liquid perfume
f) 1-5% wt of a water dispersible dyestuff on a
25 particulate, non bleaching carrier.

The invention also provides a method of cleaning a lavatory or urinal using a block of a composition in accordance with the invention.

30

In order that the invention may well be understood, the following Examples are given by way of illustration only.

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Examples

5 Toilet blocks were formed by an extrusion process in which the mixture of the components is extruded as a plastified mass through an aperture to form a billet and said billet is cut into tablets of appropriate size by means of a chain cutter.

10

The toilet blocks had the following formulation:

Example 1

wt %

15

ABS ¹	37.0
PAS ²	4.5
NaDCCA	25.0
Sodium Sulphate	13.25
20 Coloured Granules ³	2.5
Minors	to 100

ABS¹ Nansa HS 80/LPF ex Albright & Wilson

25 PAS² PAS-Empicol LZ-V ex Albright & Wilson

Coloured granules³ sodium carbonate ex Crosfield

The blocks gave a satisfactory performance and did not stain the toilet.

30

CLAIMS

1. A lavatory cleansing block comprising:
 - a) a particulate bleaching agent or precursor
5 therefore, and
 - b) a dyestuff on a particulate, non-bleaching carrier.
- 10 2. A block according to claim 1 in which the particulate non-bleaching carrier is water soluble.
3. A block according to either preceding claim, wherein the particulate non bleaching carrier comprises sodium
15 carbonate, sodium sulphate, magnesium sulphate, or sodium chloride.
4. A block according to any preceding claim in which the non bleaching carrier comprises sodium carbonate.
20
5. A block according to any preceding claim, wherein the dyestuff on a particulate non bleaching carrier b) further comprises a binder.
- 25 6. A block according to claim 5 in which the binder of b) is an acrylic maleic copolymer.
7. A block according to any preceding claim in which the bleaching agent a) is a halogen release agent.
30
8. A block according to any preceding claim in which the bleaching agent a) is selected from chlorinated cyanurates, phthalimides, p-toluenesulphonamides,

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azodicarbonamides, hydantoins, glycoluracils, amines and melamines.

- 5 9. A block according to any preceding claim in which the bleaching agent a) is sodium dichlorocyanurate (NaDCCA).
- 10 10. A block according to any preceding claim, further comprising a processing aid selected from mineral oil, silicone oil, water insoluble esters other than those derived from ethylenically unsaturated carboxyl group containing monomers, polybutene and alkoxyated alcohols.
- 15 11. A block according to any preceding claim comprising:
- a) 20-30% wt of a halogen release agent,
 - b) 2-15% wt of an oily liquid perfume, and
 - c) 1-5% wt of a water dispersible dyestuff on a
- 20 particulate, non bleaching carrier.
12. A block according to any preceding claim comprising:
- a) 1-5% wt primary alkyl sulphate
 - 25 b) 30-50% wt alkyl benzene sulphonate
 - c) 10-30% wt NaDCCA
 - d) 10-30% wt sodium sulphate, calcium carbonate or a mixture thereof,
 - e) 2-10% wt oily liquid perfume
 - 30 f) 1-5% wt of a water dispersible dyestuff on a particulate, non bleaching carrier.

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13. Use of coloured speckles within a lavatory cleansing block to indicate the presence of a bleaching agent.

INTERNATIONAL SEARCH REPORT

International Application No.

PCT/EP 99/00325

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C11D17/00 C11D3/395 C11D3/40

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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☒ Further documents are cited in the continuation of box C☒ Patent family members are cited in annex

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Date of the actual completion of the international search

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INTERNATIONAL SEARCH REPORT

International Application No
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